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A Comparative Study of MBE-Grown GaN Films Having Predominantly Ga- or N-Polarity

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Wurtzitic GaN epilayers having both Ga and N-polarity were grown by reactive molecular beam epitaxy (MBE) using a plasma-activated nitrogen source on *c*-plane sapphire. The polarities were verified by convergent beam electron diffraction (CBED). High-resolution X-ray diffraction, atomic force microscopy (AFM) imaging and transmission electron microscopy (TEM) were employed to characterize the structural defects present in the films. The different topographic features of Ga and N-polarity samples and their appearance after wet etching were correlated to the measured X-ray rocking curve peak widths for both symmetric [0002] and asymmetric [$10\bar{1}4$] diffraction. For Ga-polarity samples, the [0002] diffraction is narrower than the [$10\bar{1}4$] diffraction, while for N-polarity ones the [0002] peaks are broader than [$10\bar{1}4$]. The half width of [$10\bar{1}4$] peaks for both polarity types were in the range of 5–7 arcmin indicative of, among possibly other defects, a high density of pure edge threading dislocations lying parallel to the *c*-axis. The 1–2 arcmin [0002] linewidths of Ga-polarity samples suggest a low density of screw dislocations, which corresponds with the TEM observations where the screw dislocation density is less than 10^7 cm^{-2}. In N-polarity samples, however, the [0002] diffraction peak was typically wider than 5 arcmin, suggesting either a higher density of edge dislocations and inversion domains in N-polarity samples, or the columnar structural features in AFM images, where the effective coherence length for X-ray diffraction is drastically reduced.

Introduction The potential application of III-nitride materials for electronic devices has generated extensive research on GaN grown by MBE [1, 2]. GaN is polar along the *c*-direction, which is the most commonly used orientation for MBE growth. The quality of GaN films and performance of related device structures depend critically on the polarity of the epilayers [3]. Study of the freestanding GaN template grown by hydride vapor phase epitaxy (HVPE) has demonstrated distinct features for Ga-polarity and N-polarity GaN [4]. X-ray rocking curve linewidth of both the symmetric and asymetric reflections are narrower on the Ga-polarity side than on the N-polarity side. The overall defect density on the Ga-polarity side is more than an order of magnitude lower than that of N-polarity side. Moreover, the dependence of X-ray peak width on the dislocation structure has been studied on GaN films grown by metalorganic vapor phase deposition (MOCVD) [5], together with quantitative defect analysis by transmission electron microscopy (TEM) [6, 7].

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Recently, we have established the topological growth pattern for Ga-polarity and N-polarity GaN films by MBE through control of buffer layer parameters [8]. This allows us to assess the structural properties specific to GaN polar structure. In this work, we examine both Ga- and N-polarity GaN films using atomic force microscopy (AFM), X-ray rocking curves (ω-scan), and high-resolution TEM.

Experimental The GaN thin films were grown by MBE using a rf-plasma nitrogen source. The substrate used was *c*-plane sapphire. Both GaN and AlN buffer layers were optimized [9] and used to obtain Ga-polarity and N-polarity films through control of growth temperature and growth rate [8]. In particular, the Ga-polarity sample for TEM analysis was grown with an AlN buffer at 915 °C for 20 min and the GaN epilayer at 740 °C for 2 h. The N-polarity sample for TEM was grown with GaN buffer layer at 500 °C for 1 h, followed by GaN epilayer grown at 725 to 790 °C for 4.5 h.

The polarity of the GaN films was monitored by in-situ RHEED pattern and was established through wet chemical etching experiments, details of which can be found elsewhere [10]. It was also confirmed by convergent beam electron diffraction (CBED) on the two samples for TEM measurements. High-resolution X-ray measurements were performed on a Philips X'Pert MRD system equipped with a four-crystal Ge(220) monochromator. The instrumental broadening was ~10 arcsec. CBED, conventional and high-resolution TEM studies were perforemed using an 002B TOPCON microscope operated at 200 keV. AFM images were taken on Ga-polarity and N-polarity GaN samples before and after wet chemical etching by hot H_3PO_4.

Results and Discussions The surface morphology of Ga- and N-polarity GaN films was imaged by tapping mode AFM and is shown in Fig. 1[2]), both as-grown and after chemical etching by H_3PO_4 at ~160 °C. Distinct features of surface morphology can be observed between Ga- and N-polarity samples. The surface of as-grown Ga-polarity GaN

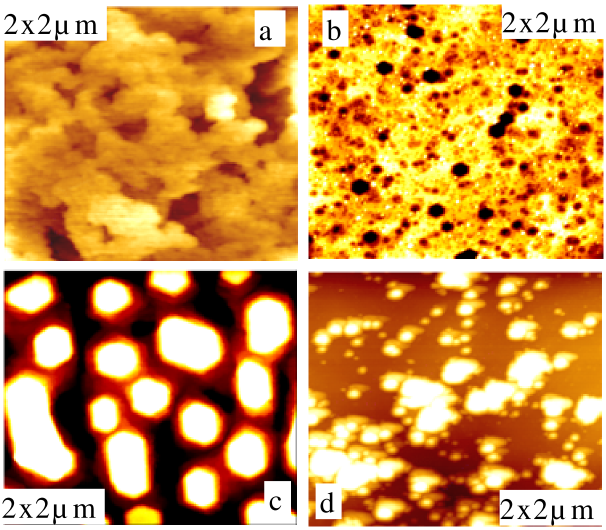

Fig. 1 (colour). AFM images of a) as-grown Ga-polarity GaN, b) Ga-polarity after etching for 5 min, c) as-grown N-polarity GaN, and d) N-polarity after etching for 30 s. Vertical scales for a) and b) are 10 nm, and for c) and d) 70 nm

[2]) Colour figure is published online (www.physica-status-solidi.com).

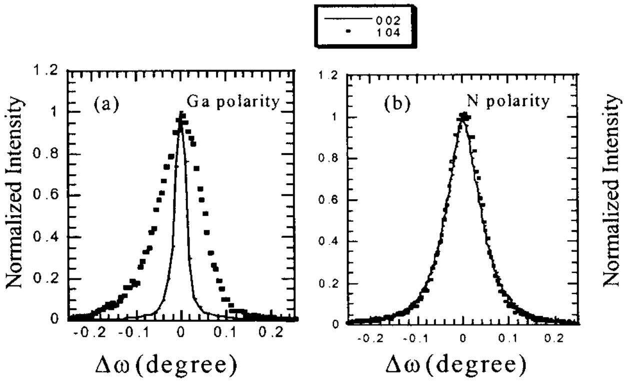

Fig. 2. Symmetric and asymmetric X-ray rocking curves for a) Ga-polarity and b) N-polarity GaN grown by MBE

(Fig. 1a) is smooth, terraced, and well-coalesced, with a root-mean-square (rms) roughness of ~3.5 nm. After exposure to etchants for 5 min, the surface of GaN remains relatively smooth, except for the presence of many hexagonal etch pits (Fig. 1b). The as-grown surface of the N-polarity film (Fig. 1c) shows isolated columns with feature size of ~0.2 μm, and a roughness (rms) of ~ 50 nm. The N-polarity film is etched quickly by hot H_3PO_4 leaving only sporadic spots (Fig. 1d) of GaN after etching for only 30 s.

Symmetric [0002] and asymmetric [10$\bar{1}$4] ω-scans are plotted in Fig. 2 for the Ga- and N-polarity samples used for TEM measurements, respectively. The Ga-polarity sample has a full-width at half-maximum (FWHM) of 1.98 arcmin for the [0002] peak, and 5.0 arcmin for the [10$\bar{1}$4] peak. The N-polarity sample shows a much larger FWHM of 5.26 arcmin for the [0002] peak, and 5.24 arcmin for the [10$\bar{1}$4] peak. Through accumulative statistics of more than 20 samples, we found that the FWHM figures of [0002] direction were between 1 and 2 arcmin for Ga-polarity GaN, and 5 and 7 arcmin for N-polarity. The FWHMs for [10$\bar{1}$4] direction were 5–7 arcmin for both Ga- and N-polarity films. This suggests that the nature and density of dislocations dominating Ga- and N-polarity films are different.

Three types of dislocations can be identified in GaN by their Burgers vectors. The edge dislocation (with Burgers vectors of ±[100], ±[110], or ±[010]) is parallel to the growth plane (c-plane) and will produce an in-plane strain which can only affect the asymmetric diffraction [10$\bar{1}$4]. The screw dislocation (with Burgers vectors of ±[001], parallel to c-axis) will produce a shear strain that can broaden the symmetric diffraction [0002]. There are also dislocations with mixed Burgers vectors (±[101], ±[011], ±[111]) which can contribute to both the symmetric and asymmetric diffractions. In order to differentiate between different types of dislocations and estimate their densities we applied conventional TEM methods since application of the diffraction contrast under two-beam condition allows for observation of a specific type of dislocations.

Shown in Figs. 3a and b are cross-sectional multi-beam bright field TEM images taken from Ga- and N-polarity GaN, repectively showing all types of dislocations. Also shown are the bright field images with a **g**-vector being parallel (**g** = 0002, Figs. 3c and d) or perpendicular (**g** = 11$\bar{2}$0, Fig. 3e and **g** = 01$\bar{1}$0, Fig. 3f) to the c-axis. For both Ga- and N-polarity films, very high dislocation density was observed (Figs. 3a and b) at the

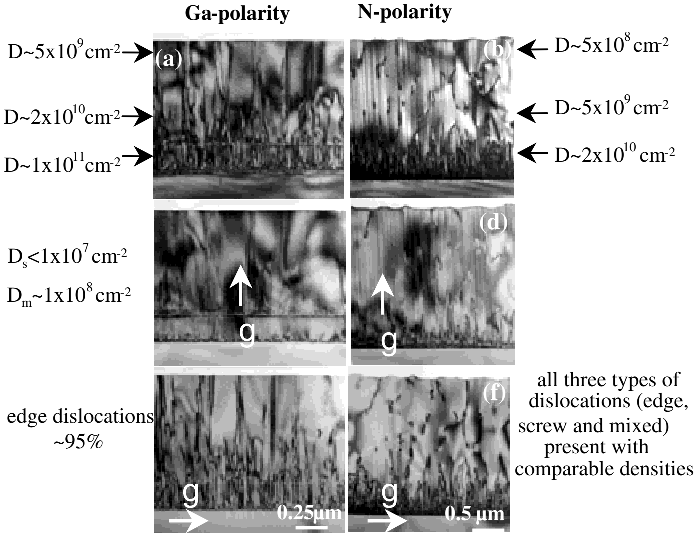

Fig. 3. TEM images showing dislocations in a) Ga-polarity and b) N-polarity GaN films. Multiple-beam dark-field images of Ga and N-polarity films with $\mathbf{g} = 0002$ are shown in c) and d), respectively, and with $\mathbf{g} = 11\bar{2}0$ in e) and $\mathbf{g} = 01\bar{1}0$ in f)

initial stage of growth (buffer layer). Some of these dislocations terminate within the buffer layer, others thread through the epilayers. The density gradually decreases with continuous growth. The total density of dislocations for the Ga-polarity sample (Fig. 3a) decreases from $D = 1 \times 10^{11}$ cm^{-2} at the buffer region to 5×10^9 cm^{-2} near the growth front. The total density for the N-polarity sample (Fig. 3b) decreases from $D = 2 \times 10^{10}$ cm^{-2} at the bottom to 5×10^8 cm^{-2} near the top surface. It should be noted that the total dislocation density of N-polarity GaN is an order of magnitude lower than that of Ga-polarity samples.

With the $\mathbf{g} = 0002$ reflection, only pure screw dislocations and mixed dislocations are visible. With $\mathbf{g} = 11\bar{2}0$, only pure edge dislocations and mixed dislocations can be observed. Therefore, it can be concluded (from Figs. 3, c, d, e and f) that for the Ga-polarity sample, edge disloactions are dominant (~95%), while the density of screw dislocations is very low ($D_s < 10^7$ cm^{-2}) and the density of mixed dislocations ($D_m = 1 \times 10^8$ cm^{-2}) is also much lower than the edge dislocations. The N-polarity sample, on the contrary, shows no dominance by any dislocation type. Instead, it exhibits an equivalent amount of all three types of dislocations. Also pronounced in N-polarity film is the large density ($D_{ID} = 1 \times 10^{11}$ cm^{-2}) of inversion domains (IDs) which occur in the Ga-polarity regions (see Fig. 3d). The density of IDs in the Ga-polarity film, however, is found to be very low (less than 10^7 cm^{-2}).

The features of Ga and N-polarity films in X-ray rocking-curve measurements can be well correlated with dislocation characteristics observed in TEM images. The narrow [0002] FWHM for Ga-polarity GaN is consistent with the very low density of screw and mixed dislocations. For N-polarity GaN, the much wider FWHM of [0002] peak agrees with the existence of a high density of screw dislocations, together with even higher density of IDs which may contribute to the broadening in a way similar to mixed dislocations. The asymmetric [10$\bar{1}$4] rocking curve peaks for both Ga- and N-polarity films have a comparable FWHM, reflecting the TEM observation of comparable edge-dislocation densities in Ga- and N-polarity films.

The symmetric X-ray diffraction peak linewidth can also be influenced by the feature size of grains or columns with respect to Ga- and N-polarity. The well-coalesced surface features of the Ga-polarity sample (in Fig. 1a) indicates less influence of broadening effects due to its large coherent length. For the N-polarity film in Fig. 1b, the columnar structure with lateral feature size of ∼0.2 μm will centainly contribute to the broadening of the symmetric [0002] peak due to the size effect predicted by the Scherrer formula [11].

Summary Distinct structural features from X-ray rocking curve measurements were found in Ga-polarity and N-polarity GaN films grown by MBE. Ga-polarity samples show much narrower [0002] linewidth than N-polarity films, while both types of films show comparable linewidths of the asymmetric [10$\bar{1}$4] diffraction. TEM images reveal the different nature and density of dislocations in Ga and N-polarity films, and are in good agreement with X-ray data. The surface morphology measured by AFM also shows different features for Ga and N-polarity films.

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